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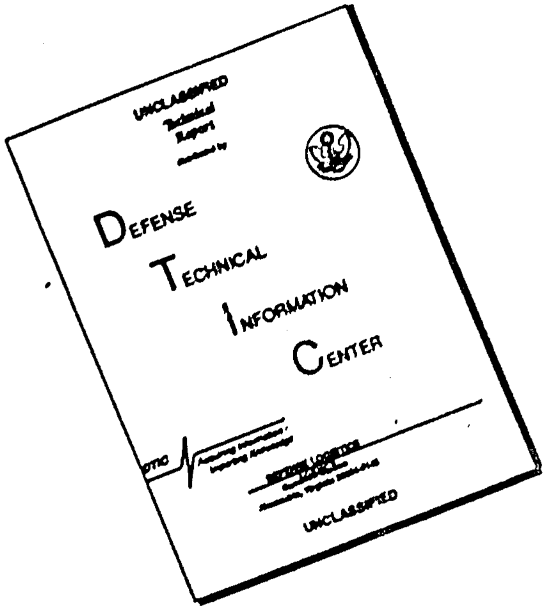

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SPECIAL REPORT

BATTELLE MEMORIAL INSTITUTE

TECHNICAL REPORT NO. 1

on

SPECIAL MEASUREMENT TECHNIQUES FOR THERMOELECTRIC MATERIALS WITH RESULTS FOR Bi_2Te_3 AND ALLOYS WITH Bi_2Se_3

to

CHIEF, PHYSICS BRANCH
OFFICE OF NAVAL RESEARCH

June 1, 1958

by

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TABLE OF CONTENTS

LIST OF FIGURES

SPECIAL MEASUREMENT TECHNIQUES FOR THERMOELECTRIC MATERIALS WITH RESULTS FOR Bi_2Te_3 AND ALLOYS WITH Bi_2Se_3

by

T. C. Harman and M. J. Logan

ABSTRACT

A new technique has been devised for the accurate measurement of the absolute values of the thermoelectric power and thermal conductivity. The method involves the use of the Peltier heat to maintain a temperature gradient across the specimen. The measurement of this gradient, the voltage across the specimen, and the a-c resistivity allows the calculation of the absolute value of the thermoelectric power and the thermal conductivity. An especially useful feature of the method is that the thermoelectric figure of merit is given in terms of the ratio of voltages. Some results for Bi_2Te_3 and alloys with Bi_2Se_3 are presented.

INTRODUCTION

Various techniques for the measurement of thermal conductivity have appeared in the literature. The most common are the comparison method and the absolute method.

In the comparison method, the thermal conductivity of the specimen is compared with that of a known standard. The sample is placed thermally in series with two standards. A temperature gradient is imposed across the standards and sample. The heat flow through the standards and the sample must be equal. Of course, good thermal contacts between the sample and the standards are necessary. Also, small-diameter thermocouple wires are used to minimize conduction of heat to or away from the specimen and standards.

In the absolute method, heat is supplied at a known rate at one end of the sample. The temperature gradient along the specimen is measured. From the measured heat input, temperature gradient, and specimen geometry, the thermal conductivity is calculated. Care is taken to eliminate extraneous heat-transfer effects.

In both of these methods, it is somewhat difficult to eliminate extraneous heat losses or gains, particularly in the case of small specimens of low-thermal-conductivity thermoelectric materials. In order to obtain more accurate measurements, an entirely new technique has been developed. However, the method can be used only for thermoelectric materials, that is, with materials having an appreciable thermoelectric figure merit, $\alpha^2/\kappa\rho$.

It is the purpose of this report to describe the technique in detail and to present some results for Bi_2Te_3 and alloys of Bi_2Te_3 with Bi_2Se_3.

SPECIAL TECHNIQUES FOR MEASUREMENT OF THERMOELECTRIC PROPERTIES

The basic circuit for measurement of thermoelectric properties by special techniques is shown in Figure 1. The arrangement consists of a sample of thermoelectric material with copper current-supply wires, Chromel-Alumel thermocouples attached to the ends, and a source of electric current. The thermocouples are placed on the ends rather than on the sides because temperature gradients can be determined more accurately if the temperature measurements are not carried out in the vicinity of a temperature gradient.

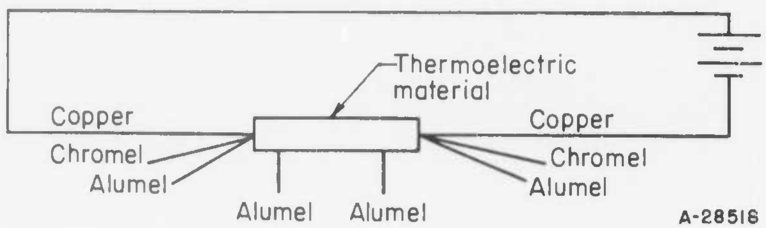

FIGURE 1. BASIC EXPERIMENTAL CIRCUIT FOR MEASUREMENT OF THERMOELECTRIC PROPERTIES

With direct current in the circuit, four principal phenomena will occur. These phenomena are the Peltier effect, Thomson effect, Joule heating, and thermal conduction. Originally there is no temperature gradient across the specimen, but at the initiation of current, a temperature gradient is developed along the bar by the thermoelectric process. If the system is adiabatic except for the introduction of the Joule and Thomson heats, which result from the current flow, then at steady state the heat transported per unit time along the bar as a result of the Peltier effect is equal to that transported in the opposite direction by thermal conduction:

$$\alpha IT = \kappa \Delta T \frac{A}{L} , \tag{1}$$

where

α is the thermoelectric power*

I is the electric current

T is the absolute temperature

κ is the thermal conductivity

A is the cross-sectional area of the specimen

L is the length of the specimen

ΔT is the temperature difference developed between the ends of the specimen.

*In the preceding simplified analysis, no distinction has been made between absolute and relative thermoelectric power. The absolute thermoelectric power of the copper leads is approximately 3 μv/°C at room temperature as compared with approximately 200 μv/°C for a typical thermoelectric material. Since copper leads are used, the relative value is within 1 or 2 per cent of the absolute values. Hence, in this report, the absolute value will be assumed.

As shown in Appendix A, the rate of Joule heating of both the specimen and lead wires of a good thermoelectric material can be made several orders of magnitude below the heat transport due to the Peltier effect. The Thomson heating effect in general is small and in addition is distributed uniformly along the specimen. Also, the Joule heating and contact resistance heating do not reverse with current. Thus, these effects can be eliminated in practice.

From Equation (1), it is seen that if the thermoelectric power is known, the thermal conductivity* can be determined from measurements of the current, temperature gradient, absolute temperature, and geometry of the specimen.

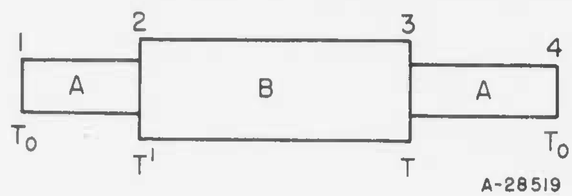

FIGURE 2. SCHEMATIC OF THERMOELECTRIC CIRCUIT

In the arrangement shown in Figure 2, consider semiconductor B in contact with metal A. Points 1 and 4 are at temperature T_o and Points 2 and 3 at temperatures T' and T, respectively. The thermoelectric power, α', of semiconductor B relative to metal A is defined by the equation

$$\alpha' = \lim_{(T'-T)\to o} \left(\frac{V_4-V_1}{T'-T} \right)$$

where V_4-V_1 is the potential between Points 1 and 4. Thus, for the experimental arrangement shown in Figure 1,

$$\alpha' = \frac{V_{\text{sample-Chromel}}}{\Delta T} \quad . \tag{2}$$

If, as in Figure 1, the thermocouples attached to the specimen are Chromel-Alumel, the thermal emf in the Chromel-sample-Chromel circuit and in the Alumel-sample-Alumel circuit give the absolute thermoelectric power of the sample by the following equations:

$$\alpha_{\text{sample}} = \frac{V_\alpha}{\Delta T} = \pm \left[\frac{V_{\text{sample-Chromel}}}{\Delta T} \right] + \alpha_{\text{Chromel}} \tag{3}$$

$$\alpha_{\text{sample}} = \frac{V_\alpha}{\Delta T} = \pm \left[\frac{V_{\text{sample-Alumel}}}{\Delta T} \right] + \alpha_{\text{Alumel}} \quad , \tag{4}$$

*This measurement technique is based on the equivalence of Peltier coefficient and αT. Recently, Russian workers[1,2] have experimentally found a small departure from equivalence in germanium. Since some of the quantities are difficult to measure with high precision, it is felt that further work is necessary before conclusions can be made.

where

α_{sample} is the absolute thermoelectric power of the sample

$V_{sample-Chromel}$ is the thermal emf of the Chromel-sample-Chromel circuit

$V_{sample-Alumel}$ is the thermal emf of the Alumel-sample-Alumel circuit

V_{α} is the absolute Seebeck potential of the sample

$\alpha_{Chromel}$ is the absolute thermoelectric power of Chromel

α_{Alumel} is the absolute thermoelectric power of Alumel

ΔT is the temperature difference between the two thermocouples attached to the sample.

The plus sign in Equations (3) and (4) is used for the case $\alpha_{sample} > \alpha_{Chromel}$, while the negative sign applies whenever $\alpha_{sample} < \alpha_{Alumel}$. Conventionally, the thermoelectric power is positive for *p*-type materials and negative for *n*-type materials.

If an independent measurement of the electrical resistivity is carried out, then the determination of the thermoelectric power as well as the thermal conductivity is possible in the same experiment. When direct current is passing through the specimen, a potential (V_T) exists between the two thermocouples used for measuring ΔT. This potential is the algebraic sum of the IR drop (V_ρ) along the specimen between the thermocouple leads and the Seebeck potential between the specimen and the specified leg of the thermocouples.

The measurement of κ, ρ, and α has been carried out with the circuit shown in Figure 1. Initially, the IR drop ($V\rho$) across the Alumel leads on the side of the specimen and the IR drop ($V\rho + V_C$) across the Alumel leads on the ends of the specimen are measured by using 60-cycle ac in a shielded room or cage.* At steady state, the total voltage across the two Alumel leads on the ends of the specimen as determined by the usual d-c null method in the two current directions is

$$+V_T(Al)^+ = +V_\rho + V_c + V_{(sample-Alumel)} + V_{Irr}(Al) \tag{5}$$

$$-V_T(Al)^- = -V_\rho - V_c - V_{(sample-Alumel)} + V_{Irr}(Al) \quad , \tag{6}$$

and the total voltage across the two Chromel leads in the two directions of current is

$$+V_T(Chr)^+ = +V_\rho + V_c + V_{(sample-Chromel)} + V_{Irr}\,(Chr) \tag{7}$$

$$-V_T(Chr)^- = -V_\rho - V_c - V_{(sample-Chromel)} + V_{Irr}(Chr), \tag{8}$$

*Because of the large Peltier effect in thermoelectric materials, accurate values of the resistivity cannot be obtained by the usual null d-c method. Values can be obtained by taking one reading of the voltage equilibrium in one direction of the current and then quickly reversing the current and taking a second reading. The average voltage is used to calculate the resistivity. However it was found here that measurements could not be made soon enough after the current was reversed.

where

$V_T(Al)^+$ is the total d-c voltage across the two Alumel leads on the ends of the sample in the plus direction of the current.

$V_T(Al)^-$ is the total d-c voltage across the two Alumel leads on the ends of the sample in the minus direction of the current.

V_ρ is the IR voltage drop across the specimen due to the resistivity of the specimen.

V_c is the IR voltage drop across the specimen due to the electrical contact resistance at the ends of the specimen.

V_{Irr} is a voltage across the specimen which is irreversible with current and hence temperature gradient. It is less than 2 per cent of the total voltage and is believed to be due in part to unequal Joule heating in the vicinity of the two dissimilar material junctions.

$V_T(Chr)^+$ is the total d-c voltage across the Chromel leads in the plus direction of the current.

$V_T(Chr)^-$ is the total d-c voltage across the Chromel leads in the minus direction of the current.

All of the above quantities are positive, changes in signs having been made in the equations.

Now, the equations for determination of the thermoelectric properties will be derived. Algebraic manipulation of Equations (3), (4), (5), (6), (7), and (8) yield for ΔT and V_α the following relationships:

$$\Delta T = \frac{V_T(Al)^+ + V_T(Al)^- - V_T(Chr)^+ - V_T(Chr)^-}{2\,\alpha_{\text{Chromel-Alumel}}} \tag{9}$$

$$V_\alpha = \pm \left[\frac{V_T(Al)^+ + V_T(Al)^-}{2} - (V_\rho + V_c) \right] + \alpha_{Al} \Delta T \tag{10}$$

or

$$V_\alpha = \pm \left[\frac{V_T(Chr)^+ + V_T(Chr)^-}{2} - (V_\rho + V_c \right] + \alpha_{\text{Chromel}} \Delta T \ . \tag{11}$$

The sign convention for Equations (10) and (11) is the same as for Equations (3) and (4). Now, the thermoelectric properties are given by the following equations

$$\alpha = \frac{V_\alpha}{\Delta T} \tag{12}$$

$$\rho = \frac{V_\rho}{I} \frac{A}{L} \ . \tag{13}$$

$$\kappa = \frac{\alpha I T}{\Delta T} \cdot \frac{L}{A} \quad . \tag{14}$$

From the above considerations, it can readily be shown that the figure of merit(3, 4), z, is given by:

$$z = \frac{\alpha^2}{\kappa\rho} = \frac{V_\alpha}{V_\rho T} \quad . \tag{15}$$

Although it appears possible to measure z with high precision by proper application of this technique, certain precautions must be taken. For example, the measurement cell which holds the specimen must be evacuated to prevent heat transfer by convection. The thermocouple and current lead wires must be long and of small diameter to minimize heat flow by conduction along the wires. More precisely, the diameter of the leads through which the current is passed must be small enough to make heat flow along them negligible, but large enough to prevent excessive Joule heating of the wires. The current and specimen geometry must be adjusted to give an adequate ΔT but not excessive Joule heating of the specimen. (Detailed calculations of the various heat-transfer effects for a typical specimen are given in Appendix A. Also, detailed calculations of the thermoelectric properties of a typical specimen are given in Appendix B.)

The following considerations give some indication of the accuracy of this new measurement technique for thermoelectric materials. The data suggest that the relative error is probably the order of 1 per cent, with systematic errors being somewhat larger.

From Equation (1), it is seen that

$$I = k_1 \Delta T \text{ and } I = k_2 V_\alpha \ ,$$

where $k_1 = \dfrac{\kappa A}{L T \alpha}$

$k_2 = \dfrac{\kappa A}{L T \alpha^2}$.

Since all factors in k_1 and k_2 are constants at a specific temperature, the ΔT and V_α will vary linearly with current. As shown in Figure 3, this variation is observed. Also, from Equation (1), ΔT should vary linearly with the distance L along the specimen for constant current, I, at a specific temperature. As shown in Figure 4, this variation is observed.

Thermoelectric properties for two Bi_2Te_3 specimens, 5b and 17a, which were measured at 300°K by these techniques, are shown in Table 1. The table illustrates the consistency of the data at the two different currents.

TABLE 1. PROPERTIES OF Bi_2Te_3 SPECIMENS 5b AND 17a AT 300°K

Specimen	α, μv/°K	κ, 10^{-2} watt/(cm)(°K)	ρ, 10^{-3} ohm-cm	z, 10^{-3}/°K	I, ma
5b	+235	1.97	1.90	1.47	5.38
5b	+237	2.00	1.90	1.47	9.70
17a	+214	2.06	1.07	2.08	20.0
17a	+214	2.05	1.07	2.09	40.0

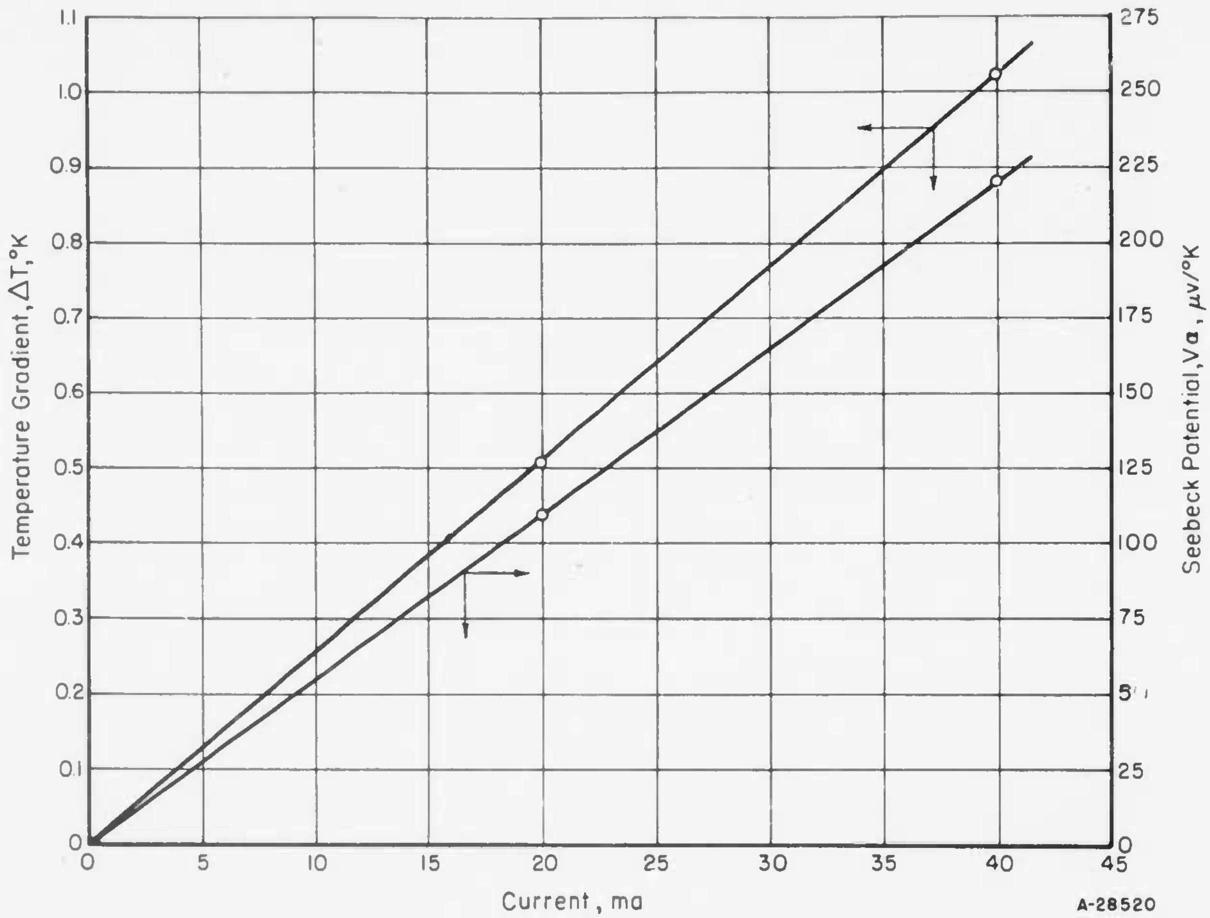

FIGURE 3. ABSOLUTE SEEBECK POTENTIAL AND TEMPERATURE GRADIENT AS A FUNCTION OF ELECTRICAL CURRENT FOR Bi_2Te_3 SPECIMEN 17a AT 300°K

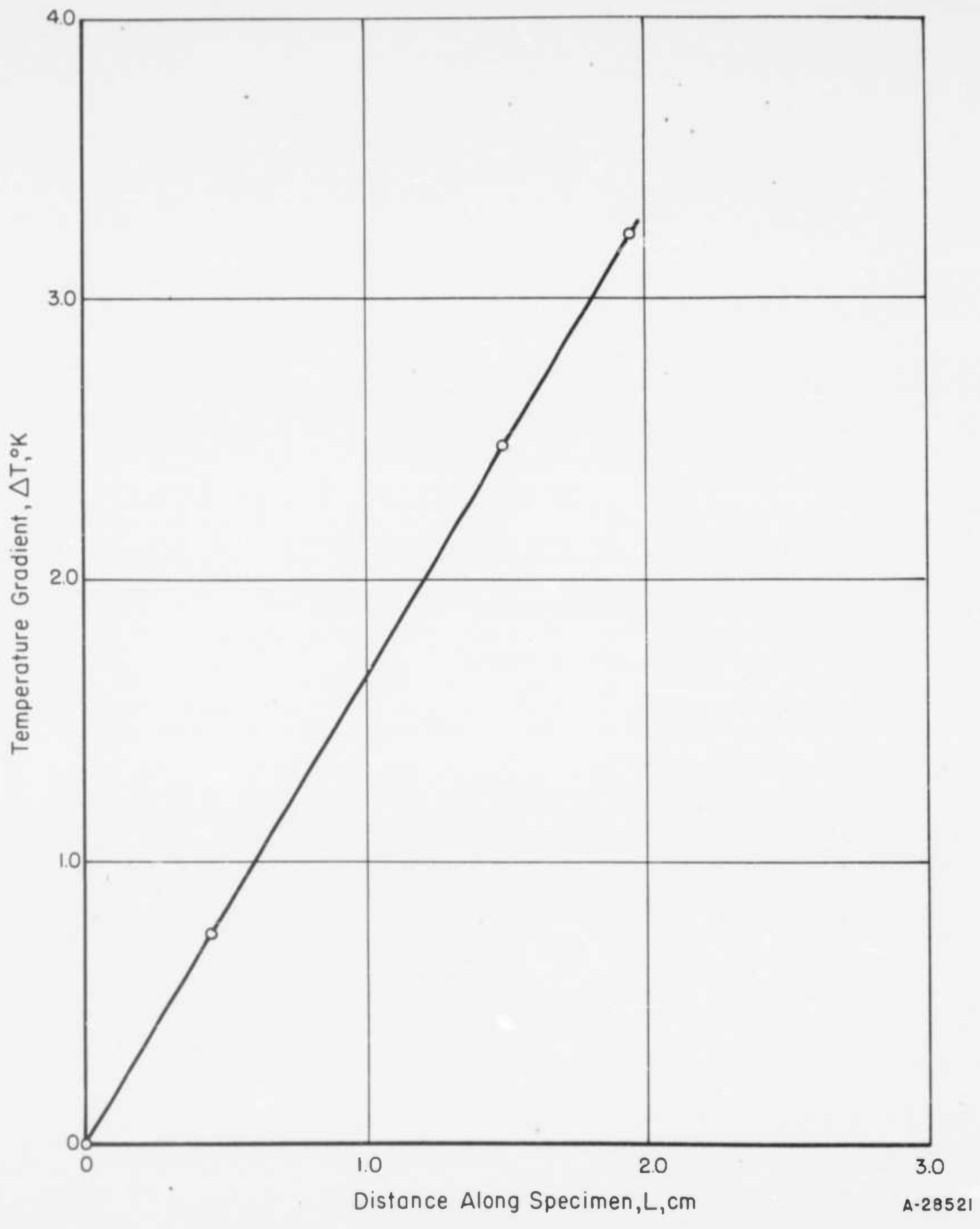

FIGURE 4. VARIATION OF TEMPERATURE GRADIENT WITH DISTANCE ALONG THE SPECIMEN FOR CONSTANT CURRENT AT 300°K

RESULTS FOR Bi_2Te_3 AND ALLOYS WITH Bi_2Se_3

The lattice thermal conductivity of Bi_2Te_3, parallel to the basal plane, has been reported by Goldsmid[5] and by Satterthwaite and Ure[6]. Figure 5 shows the results of Battelle's thermal-conductivity measurements and gives, for comparison, those by other investigators. The Battelle data were obtained by use of the measurement technique discussed in this report. It is seen that Battelle's values for the thermal conductivity of Bi_2Te_3 are in good agreement with the results of the other investigators.

The new technique can also be used for measurements as a function of temperature. Figure 6 shows the thermoelectric properties of a Bi_2Te_3-10 per cent Bi_2Se_3 alloy specimen as a function of temperature. It is seen that there is very little scatter in the data. The variations of resistivity and thermoelectric power with temperature can be qualitatively understood. The resistivity decreases with decreasing temperature due to an increase in the electron mobility. The thermoelectric power decreases because of an increase in degeneracy. However, the thermal conductivity initially decreases as temperature decreases due to the ambipolar diffusion contribution to the total thermal conductivity becoming negligible. As the temperature decreases further, the thermal conductivity increases and then remains constant. This constant region is not understood at present and will be investigated further. Additional work will be carried out on the temperature dependence of the thermoelectric properties of Bi_2Te_3 and its alloys with Bi_2Se_3.

ACKNOWLEDGMENTS

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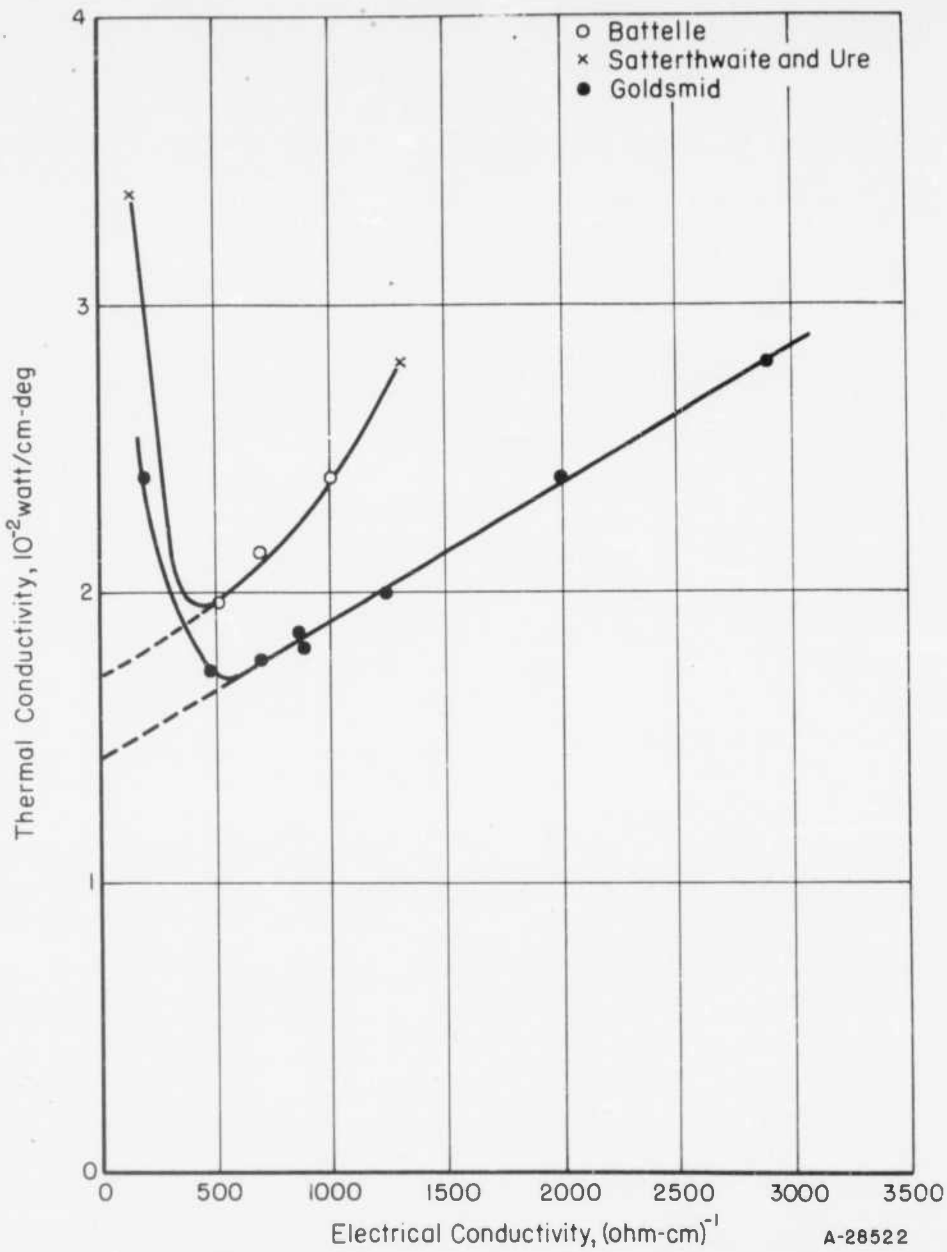

FIGURE 5. THERMAL CONDUCTIVITY (PARALLEL TO THE BASAL PLANE) AS A FUNCTION OF ELECTRICAL CONDUCTIVITY FOR VARIOUS Bi_2Te_3 SPECIMENS AT ROOM TEMPERATURE

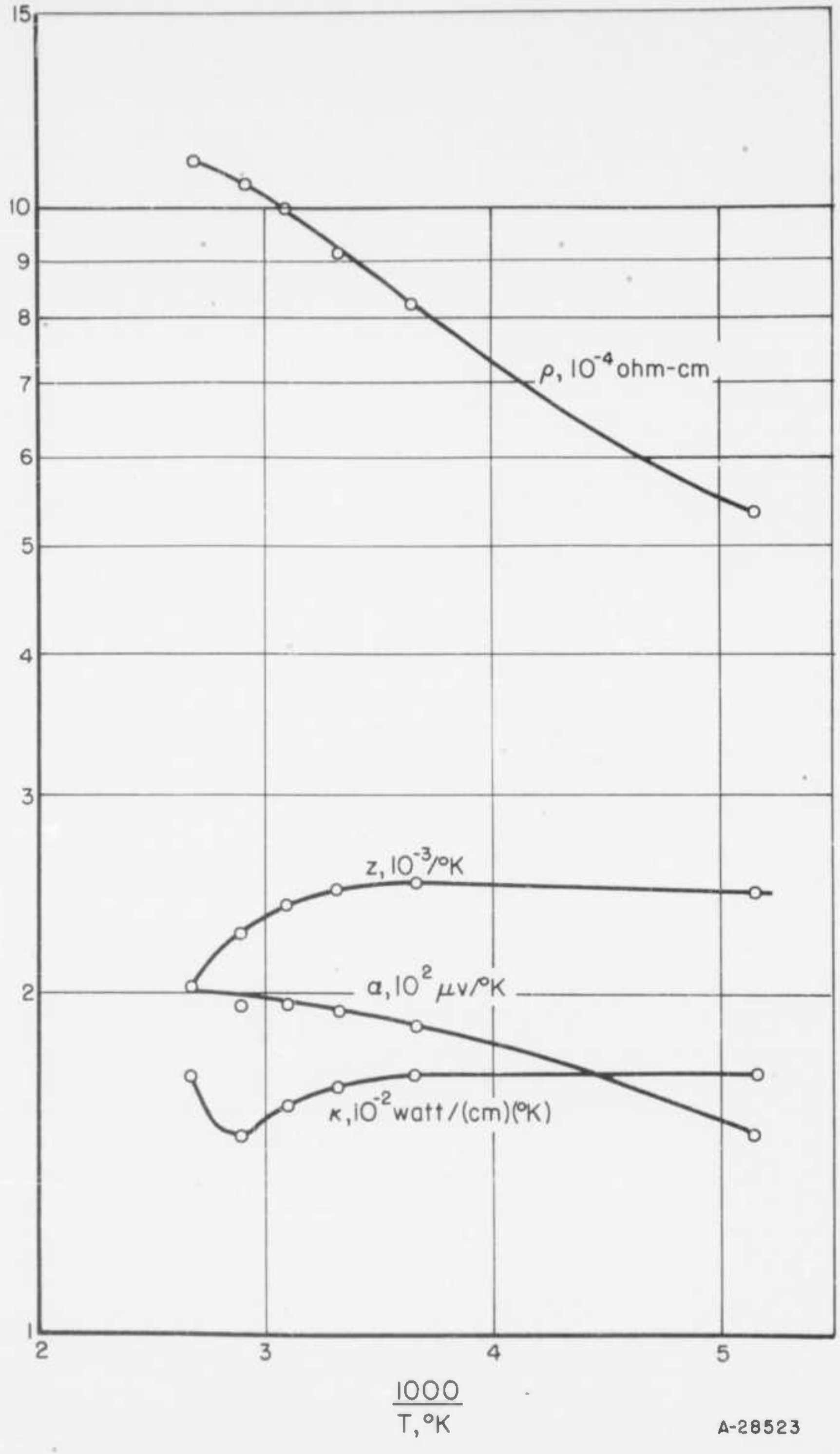

FIGURE 6. THERMOELECTRIC PROPERTIES OF A Bi_2Te_3-10 PER CENT Bi_2Se_3 SPECIMEN AS A FUNCTION OF TEMPERATURE

APPENDIX A

CALCULATION OF HEATING EFFECTS

APPENDIX A

CALCULATION OF HEATING EFFECTS

The rate of heat transfer due to a number of effects has been considered, and order-of-magnitude calculations for a recent typical specimen, 17a, are now given.

Heat will be conducted to or from the specimen through the copper current leads. The rate of heat transfer by this effect is given by

$$\frac{dQ}{dT} = \kappa_{Cu} \frac{A_{Cu}}{L_{Cu}} \left(T_s - T_a \right) , \qquad (A-1)$$

where

T_s is the temperature of the sample

T_a is the ambient temperature.

Since

$$A_{Cu} = 5.03 \times 10^{-5} \text{ cm}^2$$

$$\kappa_{Cu} = 4.18 \text{ watt/(cm)(°K)}$$

$$L_{Cu} = 25 \text{ cm}$$

$$T_s - T_a = 0.25\text{°K},$$

$$\frac{dQ}{dT} = 2.1 \times 10^{-6} \text{ watt.}$$

Heat will be conducted from one end of the sample to the other end by the Peltier effect. The rate of heat transferred by this effect is given by

$$\frac{dQ}{dT} = I T \alpha . \qquad (A-2)$$

Since

$$I = 20 \text{ ma}$$

$$\alpha = +214 \ \mu\text{v/°K}$$

$$T = 300\text{°K},$$

$$\frac{dQ}{dT} = 1.3 \times 10^{-3} \text{ watt.}$$

The rate of heat transferred from one end of the sample to the other end by thermal conduction will be given by an equation similar to (A-1),

where

$$\Delta T = 0.508°K$$

$$A = 0.227 \text{ cm}^2$$

$$L = 1.85 \text{ cm}$$

$$\kappa = 2.06 \times 10^{-2} \text{ watt/(cm)(°K)},$$

$$\frac{dQ}{dT} = 1.3 \times 10^{-3} \text{ watt.}$$

The rate of heat transfer from or to the specimen by radiation is given by

$$\frac{dQ}{dT} = A'\alpha'\sigma'\left(T_w^4 - T^4\right) , \qquad \text{(A-3)}$$

where

A' is the surface area of the sample

α' is the absorptivity of the specimen

σ' is the Stefan-Boltzmann constant

T_w is the temperature of the walls

T is the temperature of the specimen.

By using Equation (A-3), an order-of-magnitude calculation yields

$$\frac{dQ}{dT} = \sim 10^{-11} \text{ watt.}$$

There will also be Joule heating of the sample. This heating effect is calculated from

$$\frac{dQ}{dT} = I^2 \rho \frac{L}{A} . \qquad \text{(A-4)}$$

Since

$$\rho = 1.07 \times 10^{-3} \text{ ohm-cm},$$

$$\frac{dQ}{dT} = 3.5 \times 10^{-6} \text{ watt.}$$

The rate of Joule heating of one copper current lead is similarly found to be 3.4×10^{-4} watt. It is to be noted that this heating effect is only a factor of three below the Peltier heat. However, probably less than half of this heat flows into the specimen. Also, since ΔT and V_α were observed to follow a linear relationship with current, it is unlikely that this effect is playing a role. The relative effects of this heating can be reduced by using specimens that have smaller cross-sectional areas or are longer.

The transfer of heat by thermal convection has been considered. However, since the small specimen is placed in the center of a glass 6 cm in diameter and 24 cm high and the cell is evacuated to approximately 2×10^{-5} mm of Hg, this heat-transfer effect is certainly negligible.

APPENDIX B

SAMPLE CALCULATION FOR THERMOELECTRIC POWER
AND THERMAL CONDUCTIVITY

APPENDIX B

SAMPLE CALCULATION FOR THERMOELECTRIC POWER AND THERMAL CONDUCTIVITY

The actual total d-c voltages, which were measured by means of a White double potentiometer and a wall-type galvanometer, are now given for Specimen 17a. The voltage $(V_\rho + V_c)$ was measured across the Alumel leads on the ends of the specimen by means of a high-impedance voltmeter.

$V_T(Al)^+$	$V_T(Chr)^+$	$V_T(Al)^-$	$V_T(Chr)^-$	$(V_\rho + V_c)$
326 μv	307.3 μv	342 μv	320 μv	220 μv

From Equation (10),

$$\Delta T = \frac{326 + 342 - 307.3 - 320}{80} = 0.508°K .$$

From Equation (11),

$$V_\alpha = + \left[\frac{326 + 342}{2} - 220 \right] - (10 \times 0.508)$$

$$V_\alpha = 109 \ \mu v .$$

The same value is obtained for V_α by using Equation (12). The thermoelectric power is then

$$\alpha = \frac{V_\alpha}{\Delta T} = +214 \ \mu v/°K ,$$

and the thermal conductivity is

$$\kappa = \frac{\alpha ITL}{\Delta TA} = \frac{214 \times 20.0 \times 300 \times 1.85}{0.508 \times 0.227} = 2.06 \times 10^{-2} \text{ watt/cm-°K.}$$

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